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(19)

## (54) PROCESS AND APPARATUS FOR CONTACTING SOLIDS, LIQUID AND GAS

(71) We, CONSOLIDATION COAL COMPANY, a corporation organised under the Laws of the State of Delaware, United States of America, of One Oliver Plaza, Pittsburgh, State of Pennsylvania, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process and apparatus for contacting solids, liquid, and gas, and, more particularly, to a process and apparatus for reacting hydrocarbonaceous liquid with hydrogen in the presence of a solid catalyst. Still more particularly, this invention relates to a process and apparatus for reacting the hydrocarbonaceous liquid obtained by the solvent extraction of coal with

hydrogen in the presence of a solid catalyst.

Heretofore, a fixed bed reaction zone or a slurry phase reaction zone has been employed to catalytically hydrogenate a hydrocarbonaceous liquid. Both of these, however, have certain disadvantages which prevent optimum economic conditions from being attained. One of the primary disadvantages of the fixed bed reaction zone is the difficulty associated with maintaining isothermal conditions throughout the zone, while one of the primary disadvantages of the slurry phase reaction zone is the difficulty associated with effecting a substantially complete separation of catalyst from liquid products. When the above zones are employed for catalytically hydrogenating ash-containing hydrocarbonaceous liquid, e.g., hydrocarbonaceous liquid obtained by the solvent extraction of coal, they have an additional disadvantage. In the case of the fixed bed reaction zone, excessive deposition of ash on the catalyst occurs, while in the case of the slurry phase reaction zone, subsequent separation of ash particles from catalyst is difficult, if not impossible.

Fluidized bed reaction zones have been employed quite extensively in the prior art,

particularly for the treatment of a fluid reactant with a catalytic solid. The primary advantages of a fluidized bed reaction zone are the substantially isothermal conditions which may be maintained throughout the reaction zone, and the relative ease with which fluid products may be withdrawn from the reaction zone separately from the solids. Unfortunately, fluidized bed reaction zones are generally employed in two-phase systems, i.e., liquid-solid or gas-solid systems, and as yet have received little application in three-phase systems, i.e., the treatment of a liquid with a gas in the presence of a solid. The lack of application is primarily because in three-phase systems it has been found to be relatively difficult to withdraw liquid products from the fluidized bed reaction zone separately from the solids.

This invention may provide an improved process and apparatus for contacting a liquid with a gas in a fluidized bed of solids.

This invention may provide a process for economically reacting, under substantially isothermal conditions, a hydrocarbonaceous liquid with hydrogen in the presence of a catalyst in the fluidized state such that subsequent separation of catalyst from liquid products is not necessary and such that a substantial reduction in deposition of ash on the catalyst, as compared to fixed bed and slurry phase reaction zones, is obtained.

This invention may provide a process for catalytically hydrocracking a hydrocarbonaceous liquid obtained by the solvent extraction of coal in the presence of a hydrocracking catalyst in the fluidized state.

For an understanding of the present invention, reference is had to the following description and to the accompanying drawings, in which:

FIGURE 1 is an illustration, partly diagrammatic and partly cross-sectional of a dense bed, liquid phase fluidized catalytic reaction system;

FIGURE 2 is an illustration, partly dia-

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grammatic and partly cross-sectional, of a sufficient to establish and to maintain the catalyst in a liquid phase, dense fluidized bed; (b) maintaining the level of liquid in the hydrogenation zone above the level of the dense fluidized bed of catalyst; (c) passing hydrogen upwardly through the hydrocarbonaceous liquid in the hydrogenation zone, whereby a portion of the catalyst in the dense fluidized bed is carried upwardly into the liquid above the dense bed to form a liquid phase, dilute solids bed; (d) laterally diverting at least a portion of the catalyst and liquid product in the dilute solids bed such that a substantially gas-free mixture of liquid product and catalyst is passed into a cyclone separator, the cyclone separator being so positioned in relationship to the hydrogenation zone that the inlet of the separator is above the level of the dense fluidized bed of catalyst and below the surface level of the liquid in the hydrogenation zone; (e) withdrawing and thereafter recovering gas-free and catalyst-free liquid hydrogenation products from the cyclone separator; (f) reintroducing catalyst separated from the mixture of hydrogenation catalyst and liquid hydrogenation product in the cyclone separator into the hydrogenation zone; and (g) withdrawing and recovering gases from a gas disengaging zone positioned above the level of liquid in the hydrogenation zone, a portion of the gases being subsequently recovered as a hydrogen-enriched hydrocarbonaceous liquid.

The invention also relates to an apparatus for conducting reactions in a three-phase contacting zone, comprising: a reaction vessel of generally cylindrical configuration, an inlet at the bottom for a gas and liquid mixture and an outlet for the gas in the upper part thereof, a grid adapted to permit passage of the liquids and the gases, a liquid phase dense fluidized bed in the lowest portion of said vessel, a liquid phase dilute solids bed in the intermediate portion of the vessel, and a gas disengaging space in the highest portion of the vessel, a liquid-solid separator positioned within said vessel so that the inlet of the separator is above the level of said dense fluidized bed and below the level of the liquid in said vessel, means for controlling the back pressure of the gas in the gas disengaging space, and means for withdrawing substantially solids-free liquid from said separator. According to one embodiment of the invention, the separator is a cyclone separator having a cup-shaped receptacle associated therewith for receiving through the top thereof an essentially gas-free mixture of liquid and solids from said dilute solids bed and for discharging said mixture into said cyclone separator. The reaction vessel preferably has a cone-shaped bottom, and the grid is positioned between the cone-shaped section and the cylindrical section of the vessel.

In order to understand and fully appreciate

grammatic and partly cross-sectional, of a similar dense bed, liquid phase fluidized catalytic reaction system into which gas is injected;

5 FIGURE 3 is an illustration, partly diagrammatic and partly cross-sectional, of the preferred embodiment of this invention;

10 FIGURE 4 is an illustration, partly diagrammatic and partly cross-sectional, of an alternative embodiment of a portion of the apparatus of the preferred embodiment; and

15 FIGURE 5 is an illustration, partly diagrammatic and partly cross-sectional, of an additional alternative embodiment of a portion of the apparatus of the preferred embodiment.

A process, in accordance with the present invention, for contacting solids, a liquid and a gas comprises (a) passing a mixture of the liquid and the gas upwardly through the solids in a contacting zone at a velocity sufficient to establish and to maintain the solids in a liquid phase, dense fluidized bed, whereby a portion of the solids in said dense fluidized bed is carried upwardly into the liquid above said dense fluidized bed to form a liquid phase, dilute solids bed, (b) maintaining the level of the liquid in the contacting zone above the level of said dilute solids fluidized bed, (c) separately recovering gas from a gas disengaging zone positioned above the level of liquid in said contacting zone, through a gas withdrawal conduit, (d) selectively withdrawing a substantially gas-free mixture of the solids and the liquid from said dilute solids bed into a separation zone, said separation zone being so positioned within said contacting zone that the inlet of said separation zone is above the level of said dense fluidized bed and below the surface level of the liquid in said contacting zone, (e) controlling the back pressure of the gas in the gas disengaging zone whereby the liquid and solids are prevented from reaching the gas withdrawal conduit; (f) separating said withdrawn solids and liquid in said separation zone, and (g) withdrawing substantially solids-free liquid from said separation zone. According to a preferred embodiment of the invention, the liquid products and catalysts are diverted from their upward flow by flowing in a different direction, while the gas is withdrawn in a generally upward direction.

The invention also covers a process for catalytically hydrogenating a hydrocarbonaceous liquid, obtained by the solvent extraction of coal, by contacting a mixture of the hydrocarbonaceous liquid and hydrogen with a hydrogenation catalyst in a hydrogenation zone, the hydrogenation zone being maintained under conditions to yield gaseous and liquid products of hydrogenation. The process comprises (a) passing the hydrocarbonaceous liquid upwardly through the hydrogenation catalyst in the hydrogenation zone at a velocity

the present invention, a brief discussion of certain facts implicit in the invention follows. Referring to FIGURE 1 of the drawings, a reaction vessel 10 of generally cylindrical configuration with a cone-shaped bottom section is shown. The vessel 10 is provided with a bottom inlet conduit 12 and a top outlet conduit 14. Within the vessel, a grid 16 is supported by the walls of the vessel between the cylindrical and the cone-shaped sections of the vessel. The grid is adapted to permit passage therethrough of gases and liquids. Above the grid a bed 18 of particulate solids, e.g., catalyst, is provided. The particle size of the catalyst in the bed 18 is too coarse to pass through the grid 16, but is of a fluidizable size. Before any fluid is introduced into the vessel, the catalyst particles rest in a tightly packed state upon the grid. When a liquid is continuously introduced into the vessel 10 via the conduit 12, the liquid will flow upwardly through the grid 16, thence through the catalyst bed 18 and eventually out of the vessel 10 via the conduit 14. As the liquid velocity is increased, the pressure drop required to pass the liquid through the bed increases while a small but continuous expansion of the bed takes place. The pressure drop increases with liquid velocity until it is just equal to the weight of the catalyst corrected for the buoyant effect of the liquid. At this pressure drop the bed expansion has reached a point where the catalyst particles are disengaged from one another and are fully suspended in the liquid. When this state is reached, the catalyst bed is considered to reach the stage of incipient fluidization. Further increase in liquid velocity causes further bed expansion to occur and a truly fluidized state is reached. The interface between the catalyst bed in this fluidized state and the liquid phase above this bed is very pronounced, as is shown in FIGURE 1. Thus the catalyst particles are in a liquid phase, dense fluidized bed, i.e., a dense fluidized bed in a liquid phase. The catalyst particles remain discretely separate from each other and hence allow a free path to exist between them.

If gas is now injected into the vessel 10 in admixture with the liquid, the distinct separation between the liquid phase, dense fluidized bed 18 and the liquid phase above the bed tends to lose its sharpness. This may be seen by referring to FIGURE 2 of the drawings, which illustratively shows what happens when a liquid and a gas are introduced into the vessel 10 via the conduit 12. As can be seen, gas causes the loss of the sharp interface between the liquid phase, dense fluidized bed 18 and the liquid phase above it. In addition, the gas causes a portion of the catalyst particles in the liquid phase, dense fluidized bed to rise above the bed 18 and form what is hereinafter referred to as a liquid phase, dilute solids bed. In FIGURE 2 the dilute solids bed is denoted by the numeral 20. The liquid phase, dilute solids bed 20 will become more extended as the gas flow rate is increased. Likewise, the dilute solids bed will become more extended when finer size solid particles are employed.

Before liquid is withdrawn from the vessel 10 it is preferred to obtain a liquid phase substantially free of catalyst. If this is not accomplished, a subsequent vessel for separating the liquid from the catalyst must be provided. It is possible to extend the height of the vessel 10 such that eventually a substantially catalyst-free liquid phase is obtained, but the cost of doing this becomes prohibitive, particularly when working under pressure.

In accordance with embodiments of this invention, an inventory of solids is maintained in a contacting zone. A quantity of liquid is introduced into the zone to act as a fluidizing medium for the inventory of solids. The velocity of the liquid is controlled such that the velocity is sufficient to maintain the inventory of solids in a liquid phase, dense fluidized bed, i.e., a dense fluidized bed of solids in a liquid phase. The level of liquid in the contacting zone is maintained above the level of the dense fluidized bed. A quantity of gas is introduced in contact with the liquid and solids in the contacting zone. As a result, a portion of the inventory of solids rises above the dense fluidized bed and forms a liquid phase, dilute solids bed. A gas disengaging space is provided above the level of liquid in the contacting zone.

At least a portion of a separation zone is maintained within the liquid phase, dilute solids bed. Gas, liquid, and solids pass from the dense fluidized bed into the dilute solids bed. The gas continues upwardly through the dilute solids bed into the gas disengaging space from which the gas is withdrawn via a gaseous withdrawal conduit and recovered outside of the contacting zone. Liquid, however, along with a portion of the solids in the dilute solids bed, passes from the dilute solids bed into the separation zone. Gas is prevented from passing into the separation zone, as will be more fully explained hereinafter. Liquids and solids are prevented from passing from the contacting zone via the gaseous withdrawal conduit by controlling the back pressure of the gas in a disengaging space.

Within the separation zone, solids may be separated from liquid, with each being separately recovered, if desired. Generally, liquid is withdrawn from the separation zone and recovered outside the contacting zone while the separated solids are reintroduced into the contacting zone. The primary reason for being able to easily separate the liquid from the solids in the separation zone is because of the absence of a substantial amount of gas.

Preferred Embodiment  
In the preferred embodiment of this inven-

tion, hydrocarbonaceous liquid obtained by the solvent extraction of coal is catalytically hydrocracked in the presence of catalyst in the fluidized state. The extract is obtained by subjecting coal, preferably a bituminous coal such as Pittsburgh Seam coal, to solvent extraction with a hydrocarbonaceous solvent under conditions to yield an extract amounting to less than 70 percent by weight of the MAF (moisture-free and ash-free) coal. The extract is separated from the residue, for example, by filtration, and the extract is then hydrocracked with hydrogen in the presence of a dense fluidized bed of catalyst. Hydrogen-enriched hydrocarbonaceous liquid is obtained from the hydrocracking zone, a portion of which is subsequently converted to gasoline in a conventional type gasoline refining plant. The preparation of the extract is not a part of this invention. The preparation of the extract is disclosed, for example, in U.S. Patent No. 3,018,241 and U.S. Patent 3,018,242.

The following, with reference to FIGURE 3 of the drawings, is a description of the preferred embodiment of the present invention. A vessel 100 of generally cylindrical configuration with a cone-shaped bottom section is provided for confining a hydrocracking zone. The hydrocracking zone consists of a liquid phase, dense fluidized bed 102 and a liquid phase, dilute solids bed 104. The vessel 100 is provided with a bottom inlet conduit 106 and a gaseous outlet conduit 108. A grid 110 is supported by the walls of the vessel 100 between the cylindrical and the cone-shaped sections of the vessel. The grid is adapted to permit passage therethrough of liquids and gases. Above the grid a bed of hydrocracking catalyst is provided, as hereinafter more fully explained. Within the upper portion of the vessel 100 in spaced relation thereto is a separation device adapted to enclose a separation zone, the separation device consisting of a cup-shaped member 114 having a perforated top portion and a conduit 116 connecting the member 114 to a conventional type cyclone separator 118. As more fully explained below, liquid products and catalyst are passed from the dilute solids bed 104 through the perforated top portion of the member 114 and into the member 114. The mixture then flows through the conduit 116 into the cyclone separator 118. The liquid products are recovered outside the vessel 100 via the conduit 120, while the catalyst flows through a standleg 122 and into the dense fluidized bed 102.

Extract and generally a portion of the recycle liquid obtained via the conduit 120, as more fully explained hereinafter, are introduced in admixture with hydrogen into the vessel 100 via the conduit 106. The feed rate of the extract plus recycle liquid is controlled such that the upward velocity of the liquids is sufficient to maintain the hydrocracking catalysts in the form of a liquid phase, dense fluidized bed 102. Because of the gas, i.e., the hydrogen, a liquid phase, dilute solids bed 104 is formed above the liquid phase, dense fluidized bed 102. The upward velocity of the liquids will generally lie in the range of 0.0015 to 0.152 meters per second, e.g., 0.0305 meter/sec. The extract feed rate is maintained between about 16 to 2463 Kg. per hour per cubic meter of reaction space, e.g. 801 Kg./hr./m<sup>3</sup>. The proportion of hydrogen to extract is maintained between about 0.31 to 4.37 cubic meter/kilogram of extract, e.g., 0.94 cubic meter/kilogram extract. The hydrocracking zone and the separation zone are maintained under conditions employed for hydrocracking extract, generally 300 to 550°C., e.g., 440°C., and 1,000 to 10,000 psig, e.g., 3500 psig.

The catalyst may be of any of the hydrocracking catalysts usually employed by those skilled in the art. The catalyst may have a rather narrow size consist with a range of 3×100 mesh Tyler Standard screen, or even finer, e.g., 16×24 mesh, 24×48 mesh, 35×65 mesh, or 65×100 mesh. Preferably, the catalyst particle size is as small as possible, as it has been found that improved hydrocracking results are obtained as the particle size of the hydrocracking catalyst is decreased. However, as the catalyst particle size is decreased, the interface between the dense fluidized bed 102 and the dilute solids bed 104 becomes correspondingly more difficult to ascertain. The catalyst may be metals of sub-groups 5 to 8 of the Periodic Chart, preferably oxides and combinations thereof. A preferred catalyst is one containing a metal oxide or sulfide of sub-group 6 of the Periodic Chart, i.e., molybdenum, combined with a relatively minor amount of a transition group metal oxide or sulfide such as cobalt and/or nickel. The active hydrocracking metals are preferably supported on a hydrous oxide support such as alumina gel.

A preferred catalyst support for the active metals is a microbead type support generated from alumina sols by methods well known in the art. One method that may be employed to make this support is to pass the sol, under a controlled pressure head, through a plate which contains a large number of uniform size openings. The sol is then passed in the form of a descending stream of uniform size droplets through a light naphtha solution. An aqueous ammonia solution is positioned beneath the naphtha solution such that the sol droplets pass from the naphtha solution into the ammonia solution wherein the sol droplets set up to form a hard gel. The hardened droplets descend to the bottom of the ammonia solution and are then withdrawn. They are subsequently processed to prepare the finished catalyst in the conventional manner, i.e., drying, impregnation with active metals, and so on. In

this manner, hard attrition resistant catalyst particles are prepared in the desired narrow size range.

5 Catalyst, liquid hydrocracking products, and gas, i.e., hydrogen and gaseous hydrocracking products, pass from the liquid phase, dense fluidized bed 102 into the liquid phase, dilute solids bed 104. We have found that it is possible to separate the upwardly flowing liquid products and catalyst from the gas by diverting the liquid products and catalyst from their upward flow. Specifically, the liquid products and the catalyst are caused to flow laterally and then downwardly into the cup-shaped member 114, and thence via conduit 116 to the cyclone separator 118. The gas which passes through the liquid phase, dilute solids bed 104 enters a disengaging zone 124, positioned with the vessel 100 above the dilute solids bed 104. The gas is withdrawn from the disengaging zone 120 via the gaseous outlet conduit 108. It is important to note that the withdrawal of gas via the conduit 108 must be controlled such that a sufficient back pressure is provided in the disengaging zone to prevent the liquid phase, dilute solids bed from rising and passing out of the vessel 100 via the conduit 108. The back pressure is controlled, for example, by a control valve 126.

10 The absence of essentially any gas in the cyclone separator 118 makes it possible to separate catalyst particles from liquid products therein. As a result, substantially catalyst-free liquid products are withdrawn from the cyclone separator via the conduit 120, while catalysts are reintroduced into the dense fluidized bed 102 via the standleg 122.

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20 The liquid hydrocracking products, withdrawn via the conduit 120, and the gases, withdrawn via the conduit 108, are treated in any conventional manner, e.g., a portion of the liquid products may be recycled into the vessel 100, as previously mentioned, and/or a portion may be introduced into a conventional type coking zone. The gases are usually treated to recover condensable products of hydrocracking. A portion of these condensed products of hydrocracking, generally referred to as hydrogen-enriched hydrocarbonaceous liquids, may be subsequently converted to gasoline in a conventional type gasoline refining plant.

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30 Obviously, this invention is not limited to the catalytic hydrocracking of extract; but may be employed for contacting any liquid with a gas in the presence of a solid in the fluidized state. Although the specific conditions set forth in the preferred embodiment are for hydrocracking coal extract, it is relatively simple to adapt liquid and gas feed rates to a particular solid size. If desired, the single separation zone may be replaced by a multiplicity of such zones.

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40 Alternative Embodiments  
In place of the separation device shown in

45 FIGURE 3, a separation device such as the one shown in FIGURES 4 and 5 of the drawings may be employed. In order to simplify the discussion of FIGURES 4 and 5, they are numbered where possible with numbers corresponding to those in FIGURE 3 to designate corresponding parts. The separation device in FIGURE 4 consists of a cylindrical member 130 having a closed bottom end portion and a perforated lateral section, the perforated lateral section being in the lower portion of the separation device. The separation device is positioned within the vessel 100 such that the perforated lateral section is within the dilute solids bed 104. When the liquid products and catalyst are forced to flow laterally, a portion of the gas may also be laterally diverted. Thus, if in FIGURE 4 the perforated lateral section of the cylindrical member 130 is replaced by an open lateral section, gas as well as liquid products and catalyst may flow from the dilute solids bed 104 into the separation zone. It has been found, however, that if the lateral section of the cylindrical member 130 is perforated such that the perforations are larger than the catalyst particles but smaller than the gas bubbles, a substantially gaseous-free mixture of liquid products and catalyst may be passed into the separation zone. By employing this particular type of separation device, advantage can be taken of the fact that a substantial portion of the gas has the tendency to continue in the upward direction. In addition, advantage is taken of the fact that for the gas bubbles to pass through the perforated section, they must break up into smaller bubbles. This requires an amount of energy equal to the new bubble area created, multiplied by the surface tension of the liquid. As a result, substantially all of the gas flows through the dilute solids bed and into the gas disengaging zone.

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55 In FIGURE 5 of the drawings, an additional separation device is shown. The separation device consists of a generally cylindrical member 140 and a cup-shaped member 142, the cup-shaped member being provided with a standleg 144. The lower portion of the cylindrical member 140 is positioned within and in spaced relation to the cup-shaped member 142. The separation device is positioned within the vessel 100 such that the level of the dilute solids bed is above the cup-shaped member 142.

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#### WHAT WE CLAIM IS:—

65 1. A process for contacting solids, a liquid, and a gas, which comprises (a) passing a mixture of the liquid and the gas upwardly through the solids in a contacting zone at a velocity sufficient to establish and to maintain the solids in a liquid phase, dense fluidized bed, whereby a portion of the solids in said dense fluidized bed is carried upwardly into the liquid above said dense fluidized

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bed to form a liquid phase, dilute solids bed; (b) maintaining the level of the liquid in said contacting zone above the level of said dilute solids bed; (c) separately recovering gas from a gas disengaging zone, positioned above the level of liquid in said contacting zone, through a gas withdrawal conduit; (d) selectively withdrawing a substantially gas-free mixture of the solids and the liquid from said dilute solids bed into a separation zone, said separation zone being so positioned within said contacting zone that the inlet of said separation zone is above the level of said dense fluidized bed and below the surface level of the liquid in said contacting zone; (e) controlling the back pressure of the gas in the gas disengaging zone whereby the liquid and solids are prevented from reaching the gas withdrawal conduit; (f) separating said withdrawn solids and liquid in said separation zone, and (g) withdrawing substantially solids-free liquid from said separation zone.

2. The process of claim 1 wherein the solids are returned from the separation zone to the contacting zone.

3. The process of claim 1, 2 or 3, wherein in the separation zone has an outlet within the dense fluidized bed of solids such that the solids are returned from the separation zone to the dense fluidized bed.

4. The process of any one of the preceding claims, wherein the direction of flow of a portion of the liquid and solids in the dilute solids bed is changed, while withdrawing gas in a generally upward direction.

5. The process of any one of the preceding claims, wherein the direction of flow of a portion of the liquid and solids in said dilute solids bed is reversed, while withdrawing gas in a generally upward direction.

6. The process of any one of the preceding claims, wherein the direction of flow of a portion of the liquid and solids in said dilute solids bed is changed so as to pass said portion of liquid and solids into the separation zone wherein the direction of flow of the liquid and solids is changed at least one more time and the liquid and the solids are separated therein.

7. A process for catalytically hydrogenating a hydrocarbonaceous liquid, obtained by the solvent extraction of coal, by contacting a mixture of said hydrocarbonaceous liquid and hydrogen with a hydrogenation catalyst in a hydrogenation zone, said hydrogenation zone being maintained under conditions to yield gaseous and liquid products of hydrogenation, which comprises (a) passing said hydrocarbonaceous liquid upwardly through said hydrogenation catalyst in said hydrogenation zone at a velocity sufficient to establish and to maintain said catalyst in a liquid phase, dense fluidized bed; (b) maintaining the level of liquid in said hydrogenation zone above the level of said dense fluidized bed of catalyst; (c) passing hydrogen upwardly through said hydrocarbonaceous liquid in said hydrogenation zone, whereby a portion of the catalyst in said dense fluidized bed is carried upwardly into the liquid above said dense bed to form a liquid phase, dilute solids bed; (d) laterally diverting at least a portion of the catalyst and liquid product in said dilute solids bed such that a substantially gas-free mixture of liquid product and catalyst is passed into a cyclone separator, said cyclone separator being so positioned in relationship to said hydrogenation zone that the inlet of said separator is above the level of said dense fluidized bed of catalyst and below the surface level of the liquid in said hydrogenation zone; (e) withdrawing and thereafter recovering gas-free and catalyst-free liquid hydrogenation product from said cyclone separator; (f) reintroducing catalyst separated from said mixture of hydrogenation catalyst and liquid hydrogenation product in said cyclone separator into said hydrogenation zone; and (g) withdrawing and recovering gases from a gas disengaging zone positioned above the level of liquid in said hydrogenation zone, a portion of said gases being subsequently recovered as a hydrogen-enriched hydrocarbonaceous liquid.

8. The process according to claim 7, wherein the catalyst is a metal of subgroups 5 to 8 of the Periodic Chart, or an oxide of said metal or mixtures thereof and the particle size is between 8×100 mesh and 65×100 mesh Tyler Standard screen, and the hydrogenation causes hydrocracking of the hydrocarbonaceous liquid.

9. The process according to claim 8 wherein the catalyst is a metal oxide or sulfide of subgroup 6 of the Periodic Chart, combined with an oxide or sulfide of a transition group metal.

10. The process according to claims 7, 8 or 9, wherein the catalyst is on a support of the microbead type, generated from alumina sols.

11. An apparatus for conducting reactions in a three-phase contacting zone comprising a reaction vessel of generally cylindrical configuration, an inlet at the bottom for a gas and liquid mixture and an outlet for the gas in the upper part thereof, a grid adapted to permit passage of the liquid and the gases the lowest portion of said vessel containing a liquid phase dense fluidized bed, the intermediate portion of the vessel containing a liquid phase dilute solids bed, the highest portion of the vessel being the gas disengaging space, a liquid-solid separator positioned within said vessel so that the inlet of the separator is above the level of said dense fluidized bed and below the level of the liquid in said vessel, means for controlling the back pressure of the gas in the gas disengaging

space, and means for withdrawing substantially solids-free liquid from said separator.

5 12. The apparatus according to claim 11 wherein said separator is a cyclone separator having a cup-shaped receptacle associated therewith for receiving through the top thereof an essentially gas-free mixture of liquid and solids from said dilute solids bed and for discharging said mixture into said cyclone 10 separator.

15 13. The apparatus according to claim 11 or 12, wherein said reaction vessel has a cone-shaped bottom and said grid is positioned between the cone-shaped section and the cylindrical section of the vessel.

20 14. A process for carrying out a reaction with contact of solids, liquid and gas substantially as illustrated in Figures 3, 4 or 5 of the accompanying drawings and as described in the examples.

15. A process for reacting hydrocarbonaceous liquid obtained by the solvent extraction of coal with hydrogen in the presence of solid catalyst substantially as described in the examples and as illustrated in Figures 3, 4 or 5 of the accompanying drawings.

25 16. An apparatus for reacting solids, a liquid and a gas substantially as described with reference to and as illustrated in Figures 3, 4 or 5 of the accompanying drawings.

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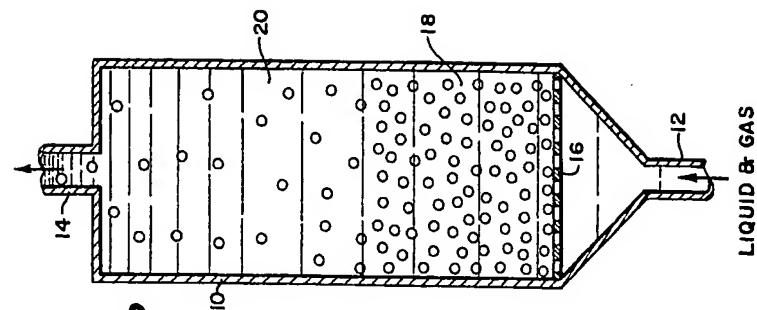


FIG. 2

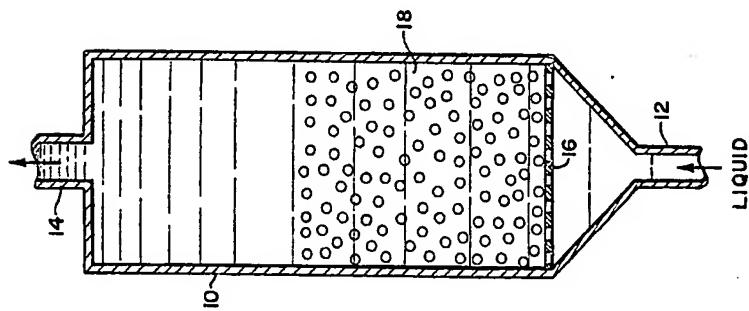


FIG. 1

